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Structure of 4-Nitrosodiphenylamine*

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Abstract. $C_{12}H_{10}N_2O$, $M_r = 198{\cdot}20$, monoclinic, $P2_1/c$, $a = 5{\cdot}932$ (1), $b = 18{\cdot}797$ (1), $c = 9{\cdot}019$ (1) Å, $\beta = 94{\cdot}00$ (1)°, $V = 1003{\cdot}2$ (2) Å^3, $Z = 4$, $D_m = 1{\cdot}30$ (1), $D_x = 1{\cdot}31$ (1) Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0{\cdot}749$ mm^{-1}, $F(000) = 416$, $T = 293$ K, $R = 0{\cdot}038$, $S = 0{\cdot}657$ for 796 observed reflections. Aromatic rings are planar and the dihedral angle between the two planes is 56·6°. There is an intermolecular hydrogen bond between N(1) and O(1 + x, $\frac{1}{2} - y$, $\frac{1}{2} + z$) of length 2·923 (5) Å.

Experimental. The title compound has wide applications in the chemical industry as a lubricant additive, an antioxidant, a stabilizer, a rubber additive, an insecticide and as a fungicide. Crystals were obtained from a 1:1 mixture of chloroform and petroleum ether. Crystal size approximately 0·1 × 0·1 × 0·4 mm, Nonius CAD-4F-11M diffractometer, Ni-filtered Cu radiation, $\omega/2\theta$ scan mode, variable scan speed with preliminary scan 1° min^{-1}, $\theta < 60°$, h 0 to 6, k 0 to 21, l −10 to 10, 1540 unique reflections, $R_{int} = 0{\cdot}031$, 796 judged significant ($|F_o| > 3\sigma|F_o|$), lattice parameters from 25 reflections ($23 < \theta < 37°$), three standard reflections ($23\bar{1}$; $\bar{1}4\bar{3}$; $21\bar{4}$) every 2000 s, 4% variation in intensity. D_m measured by flotation. No correction for absorption. Structure solved by direct methods using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Full-matrix least-squares refinement (on F) of scale factor, positional and anisotropic thermal parameters for non-H atoms. Isotropic refinement (initial positions calculated by stereochemistry and confirmed by difference Fourier map) for H atoms. Refinement converged to $R = 0{\cdot}038$, $S = 0{\cdot}657$. Total number of parameters refined 176, unit weights, $(\Delta/\sigma)_{max} = 0{\cdot}1$, final $\Delta\rho$ excursions $< 0{\cdot}3$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Program *LALS* (Gantzel, Sparks & Trueblood, 1961) was used for refinement.

The atomic coordinates with their e.s.d.'s and equivalent isotropic temperature factors are given in Table 1.‡ Bond lengths and bond angles are given in Table 2. Fig. 1 shows a *PLUTO* view of the molecule (Motherwell & Clegg, 1978).

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters for non-*H *atoms with e.s.d.'s in parentheses*

$B_{eq} = (4/3)[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + (2ab\cos\gamma)\beta_{12} + (2ac\cos\beta)\beta_{13} + (2bc\cos\alpha)\beta_{23}]$.

	x	y	z	B_{eq}(Å^2)
C(1)	7922 (6)	3549 (2)	11053 (4)	4·3 (9)
C(2)	8398 (6)	2820 (2)	10785 (5)	5·3 (3)
C(3)	6907 (7)	2416 (2)	9949 (5)	5·3 (8)
C(4)	4871 (6)	2699 (2)	9343 (4)	4·4 (9)
C(5)	4421 (6)	3423 (2)	9597 (4)	4·7 (9)
C(6)	5902 (6)	3840 (2)	10433 (4)	4·5 (1)
N(1)	9484 (5)	3927 (2)	11887 (4)	5·0 (0)
N(2)	3172 (6)	2340 (2)	8478 (4)	5·6 (9)
O	3602 (5)	1692 (2)	8316 (3)	6·3 (8)
C(1)′	9196 (7)	4631 (2)	12429 (4)	4·2 (9)
C(2)′	10882 (7)	5129 (2)	12233 (4)	5·3 (8)
C(3)′	10713 (7)	5803 (2)	12841 (5)	5·5 (4)
C(4)′	8914 (7)	5976 (2)	13631 (5)	5·0 (5)
C(5)′	7250 (7)	5483 (2)	13811 (5)	5·3 (3)
C(6)′	7380 (7)	4809 (2)	13218 (4)	4·8 (4)

Table 2. *Bond distances* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

C(1)—C(2)	1·423 (5)	N(1)—C(1)′	1·424 (5)
C(1)—C(6)	1·398 (5)	N(2)—O	1·256 (5)
C(1)—N(1)	1·354 (5)	C(1)′—C(2)′	1·391 (6)
C(2)—C(3)	1·354 (6)	C(1)′—C(6)′	1·373 (6)
C(3)—C(4)	1·396 (6)	C(2)′—C(3)′	1·386 (5)
C(4)—C(5)	1·409 (5)	C(3)′—C(4)′	1·363 (6)
C(4)—N(2)	1·403 (5)	C(4)′—C(5)′	1·372 (6)
C(5)—C(6)	1·364 (5)	C(5)′—C(6)′	1·379 (5)

C(2)—C(1)—C(6)	118·9 (3)	C(1)—N(1)—C(1)′	125·8 (3)
C(2)—C(1)—N(1)	117·7 (3)	C(4)—N(2)—O	112·8 (3)
C(6)—C(1)—N(1)	123·4 (3)	N(1)—C(1)′—C(2)′	118·5 (3)
C(1)—C(2)—C(3)	120·3 (4)	N(1)—C(1)′—C(6)′	121·4 (3)
C(2)—C(3)—C(4)	121·2 (4)	C(2)′—C(1)′—C(6)′	119·9 (4)
C(3)—C(4)—C(5)	118·2 (2)	C(1)′—C(2)′—C(3)′	119·5 (4)
C(3)—C(4)—N(2)	127·1 (3)	C(2)′—C(3)′—C(4)′	120·4 (4)
C(5)—C(4)—N(2)	114·7 (3)	C(3)′—C(4)′—C(5)′	119·7 (4)
C(4)—C(5)—C(6)	121·5 (3)	C(4)′—C(5)′—C(6)′	121·0 (4)
C(1)—C(6)—C(5)	119·9 (3)		

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‡ Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53274 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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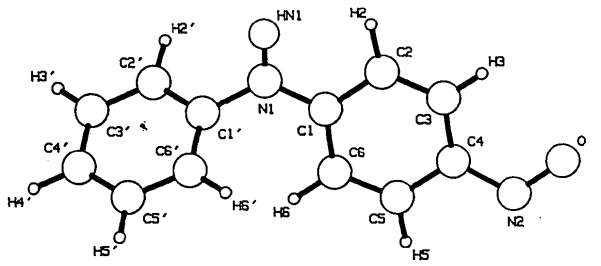

Fig. 1. Perspective view of the molecule.

Related literature. Average C—C distances of the two aromatic rings differ slightly [1·390 (5) and 1·378 (5) Å], as observed in *p*-(*p*-nitroanilino)phenyl isothiocynate (Hardgrove, Einstein & Wei, 1983). The lengths of the two C—N bonds connecting the aromatic rings are different, as observed in 6-methyl-4-(*p*-methylphenylamino)-5,6-dihydro-2-pyrone (Laidoudi, Boubekeur, Nedjar & Brianso, 1980) and in three derivatives of 9,10-anthracenedione (Foitzik, Paulus & Haase, 1986; Foitzik, Paulus & Quotschalla, 1987).

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Structure of Myrotoxin B Hydrate

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Abstract. Myrotoxin B hydrate ethyl acetate solvate, $C_{29}H_{36}O_{12}.C_4H_8O_2$, $M_r = 664{\cdot}71$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}911$ (1), $b = 15{\cdot}366$ (2), $c = 22{\cdot}548$ (3) Å, $V = 3434{\cdot}1$ (7) Å^3, $Z = 4$, $D_x = 1{\cdot}286$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 0{\cdot}81$ mm^{-1}, $F(000) = 1416$, $T = 295$ K, final $R = 0{\cdot}048$, $wR = 0{\cdot}063$ for 3131 independent reflections with $F_o > 3\sigma F_o$. In the macrocyclic ring the two hydroxyls of the tetrahydropyranyl ring are *cis* to each other with the hydroxyl on C(12)′ axial to the ring. The solvate molecules pack in channels parallel to the *a* axis, and there is an intermolecular hydrogen bond between the two hydroxyls with the axial hydroxyl acting as a donor [H···O = 1·83 (5), O···O = 2·735 (7) Å, and ∠O—H···O = 170 (3)°].

Experimental. A clear colorless 0·30 × 0·45 × 0·64 mm data crystal was provided by Bruce Jarvis of the University of Maryland. Automated Siemens *R*3*m*/*V* diffractometer with incident beam monochromator. 25 centered reflections within $39 \le 2\theta \le 78°$ used for determining lattice parameters. $[(\sin\theta)/\lambda]_{max} = 0{\cdot}59$ Å^{-1}, range of *hkl*: $0 \le h \le 11$, $0 \le k \le 18$, $0 \le l \le 25$. Standards 4,0,0; 0,8,0; 0,0,10, monitored every 60 reflections with random variation of 2·7% over data collection, $\theta/2\theta$ mode, scan width $[2\theta(K\alpha_1) - 1{\cdot}0]$ to $[2\theta(K\alpha_2) + 1{\cdot}0]°$, scan rate a function of count rate (2·0° min^{-1} minimum, 15·0° min^{-1} maximum) in ω, 3482 reflections measured, 3300 unique, $R_{int} = 1{\cdot}1\%$, 3131 observed with $F_o > 3\sigma(F_o)$. Data corrected for Lorentz, polarization and absorption effects, max. and min. transmission 0·84 and 0·76. Structure solved by direct methods. The least-squares refinement used program *SHELXTL* (Sheldrick, 1980). $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$, $g = 0{\cdot}00023$. Secondary-extinction parameter $p = 0{\cdot}0028$ (5) in $F_c^* = F_c/[1{\cdot}0 + 0{\cdot}002(p)F_o^2/\sin(2\theta)]^{0{\cdot}25}$. There were 448 parameters refined: atom coordinates, anisotropic thermal parameters for all non-H atoms, H atoms included

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